Processing and property evaluation of Iron oxide Powder Reinforced polymer composite

Thesis submitted in partial fulfillment of the requirements for the degree
of

Master of Technology

in

Metallurgical and Materials Engineering

Submitted By

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Certificate

This is to certify that the summer report entitled, "**Processing and property evaluation of iron oxide powder reinforced polymer composite"** submitted by **Debasmini Prusty** bearing **Roll No. 213MM2486**, **Department of Metallurgical and Material Engineering, National Institute of Technology Rourkela,** is an authentic work carried out by her under my supervision and guidance. I certify that the work has not been submitted elsewhere for any other report.

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Date: **Debasmini Prusty**

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ABSTRACT

Composites are the materials consisting of two or more chemically dissimilar constituents, on a macro-scale, having a distinct interface separating them. The discontinuous phase is usually is called as reinforcement, whereas the continuous phase is termed as the matrix. Various kinds of polymers and polymer matrix composites reinforced with metal particles have a large variety of engineering applications such as electrodes, heaters, with thermal toughness at high temperature, etc. The addition of inorganic fillers into polymers for profitable applications is primarily intended at the cost reduction and stiffness improvement. In the present study, an attempt has been taken to fabricate iron oxide powder reinforced epoxy matrix composite and evaluate its properties for different possible applications. Ten number of samples with different Fe_2O_3 and epoxy composition viz. 1:9, 2:8, and 3:7, were prepared. The samples were also dipped in sea water to study the moisture absorption and other properties. The hardness study is performed by Vickers hardness tester (LECOLM 248AT). The compressive strength of all the cylindrical samples was evaluated with INSTRON 1195. The density was measured; the wear behavior was evaluated by a pin on disc wear testing machine (DUCOM). The dielectric behavior of the composites is also studied.

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CHAPTER - I

INTRODUCTION

1.1INTRODUCTION

Composites are materials consisting of two or more chemically distinct ingredients on macro-scale, having a different interface separating them. One or more irregular phase is therefore embedded in a matrix of the material having different properties to fabricate a composite. The reinforcement is typically harder and stronger than the continuous phase. The reinforcing phase can either be tough or non-fibrous (particulates) in nature. The reinforced material is embedded in or bonded to a matrix. In this shape together fibers and matrix remain their physical and chemical identities. In universal fibers are the chief weight transport members at the same time as the matrix keeps them in the preferred position and direction acts as a weight transport intermediate between them, and protects them from environmental damages [1]. Composites have many uses in automobiles, spacecraft, satellites, ships, submarines, sporting goods, chemical processing equipment, aircraft, helicopters and civil infrastructure, and there is the probable for ordinary utilize in medicinal prosthesis and microelectronic devices. Composites are important materials for the reason that of their glow heaviness high specific potency and stiffness, outstanding fatigue struggle and exceptional corrosion confrontation compared to most common metallic alloys such as steel and aluminum. Other advantages of composites include the ability to fabricate, directional mechanical properties, high dimensional strength. It is the combination of outstanding physical, high stiffness, strength and fatigue resistance, etc. The material and automatic distinctiveness can further be customized by the addition of a solid filler stage to the medium body through the composite preparation. Explicit fillers (additives) are mixed to improve and change the quality of composites. The fillers play a major role in forming the properties and performance of particulate reinforced composite material.

1.2 Classification of Composite

- Composites can be very tough and stiff, light weight so ratios of strength-to-weight and rigidity are several times larger than steel or aluminum.
- Toughness is often greater. Fatigue properties are usually better than for ordinary engineering materials.
- Composites can be designed that do not corrode like steel

- Composites are divided into three parts based on their matrix resources

- Ceramic matrix composites
- Metal matrix composites

- Polymer matrix composites

1.3 Applications of Reinforced Plastics

- This region of the composites industry is characterized by the employ of luxurious high-performance resin systems and high strength, high in flexibility fiber reinforcement. Composites are needed most in the aerospace industry, together with military and cost-effective aircraft.

1.4 Advantages of Composites

Composites are important materials because of their frivolity, elevated detailed strength and rigidity, outstanding fatigue confrontation and exceptional corrosion confrontation compared to most common metallic alloys such as steel and aluminum.

1.5 Disadvantages of Composites

Composites are diverse.

Most of the engineering materials are homogeneous in nature, but composites are directional nature, so they are heterogeneous materials

Composites are highly anisotropic and heterogeneous.

The power in composites differs as the path along which we calculate changes (most engineering structural materials are isotropic). As a consequence, all additional properties such as stiffness, thermal and electrical conductivity, thermal expansion and creep conflict is also anisotropic. The association among stress and strain (force and deformation) is much more complex than in isotropic material.

CHAPTER - II

LITERATURE REVIEW

2. Literature Review

2.1 Introduction

The purpose of the literature review is to supply background in sequence on the issues to be considered and to highlight the need and significance of the present study. The topics include brief review:

- On fiber/ particulate reinforced polymer composites
- On multiphase hybrid composites
- On mechanical properties of composites

2.2 Fiber reinforced polymer composites

Fiber resistant polymer composites are now calculated as a vital class of engineering materials. They offer exceptional mechanical properties, exceptional flexibility in design capability and ease of fabrication. Supplementary compensation comprises glow weight, corrosion and shock resistance and outstanding fatigue strength. It consists of fibers entrenched in or bonded to a polymer matrix with different interfaces between the two constituent phases. The fibers are typically of high potency and modulus and serve up as the principal load carrying members. The medium acts as the load transfer average between fibers and in less ideal cases where loads are complex, the matrix may even contain to partially bear loads. The matrix also serves to protect the fibers from environmental damage before, during and following composite dispensation.

2.3 Particulate filled polymer composites

Rigid particulate fillers consisting of ceramic or metal particles and filament fillers made of glass are existing being used these days to noticeably get better the wear resistance even up to three orders of magnitude. A mixture of polymers and polymer matrix composites reinforced with metal particles have a broad variety of industrial applications such as heaters, electrodes, composites with thermal durability at high temperature etc. These manufacturing composites are favored due to their low density, high corrosion resistance, ease of fabrication, and low cost. Similarly, ceramic filled polymer composites have been the matter of wide-ranging investigate in last two decades. The addition of inorganic fillers into polymers for profitable applications is primarily aimed at the cost reduction and stiffness enhancement.

2.4 Mechanical properties of composites

The mechanical properties of a thermoplastic polymer like Polypropylene (PP) have been modified by adding various mineral fillers such as talc and calcium carbonate. It has also been shown that such filler particles increase Young's modulus of PP, yet causing the decrease of the strength and the stiffness PP can also be resistant with short glass fibers (SGF) to improve the stiffness and the fracture toughness (1, 2). However, long glass fibers (LGF) are used more often as reinforcement since it is known that longer fibers with the same fiber diameter (i.e. with higher fiber aspect ratio) supply higher rigidity, tensile strength and roughness compared to shorter ones(3). lately it has been experimental that by incorporating filler particles into the medium of fiber resistant composites, synergistic property may be achieved in the form of higher modulus and reduced material expenses, yet accompanied with decreased force and impact toughness [4, 5]. As already mentioned, such multi-component composites have a matrix phase resistant with a fiber in addition to packed with particulate matters are named as hybrid composites.

2.5 Reviews on Composites and Epoxy resin

M. Golestaneh, G. Amini, G.D. Najafpour and M.A. Beygi [6] showed that there has been significant increase in use of glass fiber reinforced composites as structural resources in naval mine counter compute surface ships.

Jia et.al carried out similar examination on the sport and transport distinctiveness of carbon fiber reinforced polymer composites (in distilled water lubricated) dry-sliding wear against stainless steel. Scanning electron microscopy (SEM) was used to observe complex microstructures and modes of breakdown. The characteristic constituent substance tells the transport pictures on the stainless steel were experimented with X-ray photoelectron spectroscopy (XPS). It was established that all the composites grasp the lowered friction coefficient and showed much enhanced wear resistance beneath water lubricated descending adjacent to stainless steel than below dry sliding. The spoiled of composites were examined by plastic deformation, scuffing, micro-cracking, and spelling under both dry- and water lubricated circumstances. Such plastic deformation, scuffing, micro-cracking, and spelling, however, are considerably abated beneath water-lubricated circumstance. XPS examination conformed to that the transport of composites onto the complement ring surface is considerably caught up under water lubrication. The transport film had lesser result on the tribological performance of composites below water lubricated form than that under dry sliding, since under water-lubricated circumstances the cooling and boundary lubricating property of the water medium conquered the tribological behavior [7].

Imran Oral (8) studied to analyses the composites of epoxy resin by ultrasonic method. The ultrasonic wave velocities of composites were measured with pulse echo method at room temp by a flaw Detector. The substance is epoxy resin. The importance of the poison “ratio, acoustic impedance, and elastic constants of the calculated standards of the densities and together longitudinal and shear ultrasonic wave velocities. Different type of coagulants and dosages for recycling of marble waste for the production of ER composites.

C.ATZENI, L.Mussidda and U Sanaa (9) investigated the use of fly ash filler mortar containing epoxy resin as binders as a replacement for quartz flour used for application in paving for industries, airports and heavily trafficked roads. The results indicate that the specimens containing fly ash display better mechanical properties in comparison with the quartz filler ones of the shorter curing times, at the same time as at longer curative instance mechanical presentation is equivalent.

Pradeep k. rohatgi, Takuya Matsunaga and Nikhil gupta. (10) Studied the density of composite can be decreased by the addition of hollow filler having mechanical properties called synthetic foams, while improves mechanical properties. It is a waste byproduct of coal combustion. The result of this process is a graded structure containing a gradient in the cenospheres volume fraction along sample height.

M Mustafizur Rahman Akhtar Islam (11) studied the properties of three type of masonry mortar, they are Portland cement mortar, polymer cement mortar and polymer mortar of various composition. The result of binder content cement or epoxy o CM, PCM or PM has been explored and studied. Mechanical properties similar to compressive, tensile and flexural strength, physical properties like water uptake, chloride ion permeability, morphological property like porosity and thermal property like coefficient of thermal expansion were measured in this study. Mechanical properties of masonry mortar enhanced by the accumulation of epoxy resin. The intrinsic properties calculated for dissimilar types of mortars are different depending on the composition and epoxy content.

Valeria Corinaldesi, GiacomoMarconi, Tarun.R.Naik(12) studied the increasing popularity of fiber reinforced polymer (FRP) composites. The employ of FRP is a information for rising flexural and shear strength of scarce resistant concrete members. Tensile and bond test were approved out carbon FRP distorted rods for application as near surface mounted reinforcement. Tensile and bond tests on FRP deformed rods for purpose as NSM reinforcement were accepted out using coupon size specimens. These two test gave effective results.

Huseyin Yilmaz Arunta, MetinGuru, MustafaDayi, LikerTekin, (13) studied the usability of waste marble dust (WMD) as an additive matter in blended cement. Waste marble dust added cement have obtained by intergrading WMD with Portland cement clinker. It produced mortar prism with the obtained cement. Strength tests have been accepted out on the mortar specimen. Consequences showed that only 10% WMD can be damaged as an additive matter and cement manufacturing.

V.m.malhotra, p.s.valimbe(14, 15) studied the property of fly ash and bottom ash on the frictional activities of composites. It is predictable that regarding 63million tons of fly ash and concerning 17million tons of bottom ash are formed every year. So to enhance its utility, it is used in ultra-light heaviness aggregates, highway and street building, construction bricks or tiles, roofing or concrete tiles, pipe building, synthetic reefs for aquatic wildlife habitation and aluminum fly ash composite resources. Fly ash, bottom ash and sulfate loaded scrubber sludge are used as friction modifiers and additives for automotive frictional composites. The coarse materials like Al_2O_3, Fe2O3, MgO, ZnO etc. are incorporated in a composite to manage the frictional individuality. Integration of <106 micro meter fly ash or bottom ash particle manage the mechanical, structural and frictional characteristics of brake composites as estimated by SEM and DMA. The SEM photographs of the fly ash showed the particle to be largely spherical with the bulk of the particles being and the range of <3.5 micro meter. If the fly ash or bottom ash particle which are incorporated into frictional composites makes the materials too rigid or too soft or induce tendency to flake then they are not suitable because they would increase wear. The frictional behavior of composites are determined by FAST technique. From microscopy, mechanicals well as frictional tests, following were concluded. One is the scrubber mud particles in frictional composites are highly tough particles having bunch like structure. Another is the addition of fly ash or bottom ash into composite materials formulated from resin, slag fibers and modulus of the composites.

V.K.SRIVASTAVA, A.G.PAWAR (16, 17)studied the activities of solid element corrosion of glass fiber resistant fly ash packed epoxy resin composites. Fiber reinforced plastic (FRP) composites have different applications in automobile, aerospace and marine.FRP composites are practical to, fan exit-guide vanes, inlet cone and other parts of structures in a turbofan engine for lightening an engine. The property of fly ash stuffing, impingement angle and particle velocity on the solid element erosion behavior of E-glass fiber reinforced epoxy composites were studied. Erosive wear behavior was studied at different impingement angles from 30 degree to 90 degree and at three different velocities 24, 35 and 52m/sec. The test materials used are epoxy resin and hardener HY-951.The most generally used technique for estimating impact velocity of the erodent particle is double disc method. Micro-hardness test is conducted on Shimadzu micro hardness tester. Indenter is completed of diamond in the shape of square based pyramid with an integrated angle of 136 degree among opposite faces. The test shows the micro-hardness of GFRP and fly ash filled GFRP reduces with the increase of load. Erosive efficiency is used to describe erosion mechanism. The erosive wear of GFRP composites exclusive of any filler material is much superior than that of fly ash filled GFRP. Hardness of GFRP is more than that of fly ash filled GFRP.

T.CHAOWASAKOO, N.SOMBATSOMPOP (18)studied the mechanical and morphological properties of fly ash/epoxy composites through resources of conservative thermal and microwave remedial techniques. Conventional thermal and microwave curing techniques are used to treat fly ash/epoxy composites. The mechanical and morphological properties of composites are estimated in this techniques. The tensile and flexural module of the composites are increased with increase in fly ash content while the consequence becomes reverse for tensile, flexural and impact strengths and tensile strain at break. Ash residues that are produced at the boiler outlet of the plants including fly ash and bottom ash are wastes of coal-fired power plants. Fillers materials are used in the epoxy resin include inorganic, organic and ceramic materials, improve the processing and product performance and to reduce cost of epoxy resin. Fly ash particle are chemically treated and introduced in epoxy resin and the composites are cured by conventional thermal and microwave cures. Raw material used is epoxy which is a Diglycidyl ether of Bisphenol-A and hardener 3-amino-methyl-3, 5, 5-trimethycyclohexy-amine.N-2-3-amino propyltrim ethoxysilane is used as a chemical coupling agent for FA surface treatment. Tensile properties of the composites are tested using a universal testing machine. Fly ash mixed epoxy composite samples after impact test are examined with a JOEL SEM apparatus at 17KV accelerating voltage

Leng Jinfeng, JiangLongtao, WuGaohui, TianShoufu(19) studied the effect of graphite particle reinforcement on dry sliding wear of Sic/Gr/Al composites. Metal matrix composites have been worn I the aerospace, automotive and aircraft industries since they acquire lots of potential compensation over monolithic substance having higher specific strength and rigidity,, inferior co-efficient of thermal expansion, superior thermal conductivity, higher wear resistance. The 40%SiC/5%Cr/Al composites with various sizes graphite addition are fabricated by squeeze casting technology and their friction and wear characteristics are investigated. The addition of graphite, the friction co-efficient of composites decreases and wear resistance is increased by 170 to 340 times. Squeeze casting equipment is successfully used to fabricate SIC/Gr/Al composites and the consequence of graphite on friction co-efficient because fine wear resistance of the resulting composites is investigated. Dry sliding wear tests are performed on the samples made of SiC/Gr/Al composites and SIC/Al composite to find the material properties. Tensile strength of SiC/Gr/Al composite are decreased with the addition of graphite particle. Scanning electron microscope attached with energy dispersive X-ray spectroscope is utilized to characterize morphology, compositions of wear surfaces. The development of wear resistance is credited to the enhancement of reliability of lubrication tribo-layer collected of a complex mixture of iron oxide and graphite.

K.Srinivas,M.S.Bhagyashekar(20) studied the wear behavior of epoxy hybrid particulate composites. The RT cured epoxy composites used to position cure cycle containing particulate Gr,SiC of length 25mm and diameter 10mm are the pin specimen and EN31steel is the compact disk of mechanized pin on disc wear tester. Particulate filled epoxy is used for non-structural purpose such as polymer bearings, tools completed by fast prototyping underneath fills and encapsulates. The reinforcing fillers collection from particulates of metals, carbides, oxides, nitrides, and solid lubricants such as graphite, mica, PTFE and Nano sized fillers .Addition of metallic fillers improves wear resistance of epoxy. Micro-particulates of SiC size less than 60 micro meter and graphite particulates of size 20 micro meter are the reinforcement. Measurement of wear and friction tests are conducted on a computerized magnum make pin on disc type wear tester under dry sliding condition.SEM is studied on the worn surface of composite using Joel make 5600LV scanning electron microscope. The epoxy particulate composite containing SiC/Gr exhibits lower specific area but epoxy hybrid particulate composite containing SiC/Gr exhibits lowest specific wear/higher wear resistance.

C.RajeshChandra,T.R.Ravikumar,N.Mohan,C.R.Mahesha(21)studied the mechanical and three-body abrasive wear actions of Nano fly ash/ZrO2 packed polyimide composites. Co-efficient of friction and the wear rate of polymer composite can be improved if the polymers are combined with traditional tribo-fillers. Polymers and their composites are used in tribological application since of light heaviness, outstanding strength to heaviness ratios, confrontation to corrosion, non-toxicity, easy fabrication, design flexibility, self-lubricating properties, good co-efficient of friction and wear resistance. A small number of examples of method used in tribological applications are cams, brakes, rollers, wheels, bearing liners, seals, clutches, bushings, transmission belts, pump handling industrial fluids, pipes carrying contaminated water and chute liners abraded by coke. Effect on the mechanical performances of composites depends on their property, shape, dimension, size and combined degree, plane distinctiveness and application. Polyimide is a polymeric plastic fabric engineered for long expression presentation at extremely high temperatures in surplus of 250 degree centigrade for extended periods of time. Polyimide produced from ABR organics Ltd. is measured as the matrix material. Fly ash from Nayeli Lignite Corporation and ZrO2 from LOBO chemicals are used as potential fillers materials'-ray diffraction uses x-rays to examine the crystalline nature of resources by calculating the diffraction of x-rays starting the planes of atoms within the materials. Hot compression molding technique is used for the preparation of samples. The tensile test is conducted on a universal testing machine and the hardness test is done using Shore-D hardness tester. The conclusion is the addition of Nano fly ash/ZrO2 particle increases mechanical properties of polyimide composites.

S.R.Chauha, Sunil Thakur (22)have studied the property of element size, element loading and sliding detachment on the friction and wear properties of cenospheres particulate packed vinyl ester composites. The evaluation of the effects of particle size, element loading and sliding detachment on the friction and wear behavior of vinyl ester composites sliding adjacent to hardened ground steel on a pin-on-disc wear testing machine. The polymer composites materials provide unique mechanical and tribological properties united with a small specific weight and a high resistance to degradation. These materials are highly used and have high demand in naval ships, warplanes and sea vehicles because of its own density and good strength. The polymer composites have high surface energy, so the submicron particles have influence on wear behavior of polymer which may depend on the quantity content, size, shape of sub-micron and co-efficient of friction. The experiments are in make use of the co-efficient of friction and wear rate as a purpose of parameters such as sliding distance(5000,25000m) at two dissimilar functional loads of 10N,70N and sliding speeds 1.9m/s and5.7m/s. The experimental materials are vinyl ester resin, hardener, accelerator and filler materials that are cenospheres. The submicron particles and micro particles are able to improve the mechanical and wear resistance of the cenospheres filled vinyl ester composites. The hardness, compressive strength and flexural strength is enlarged linearly with reduce in the cenospheres particle size in vinyl ester composite which is owing to physically powerful bonding among particles and the matrix.

R.V.Kurahatti, A.O.Surendranathan, A.V.Ramesh,C.S.Wadageri,V.AuradiandS.A.Kori(23) studied dry sliding wear behavior of epoxy reinforced with Nano ZrO2 particle. An experiment was conducted to investigate the matrix properties by introducing Nano size ZrO2 filler into epoxy resin. Polymer composites are increasingly used as structural capital I aerospace, automotive and chemical industry because they provide potential lower weight alternative to traditional metallic materials. The addition of nano-ZrO2 particles increases flexural modulus and flexural strength of epoxy, the wear properties doesn't relate through these stationary mechanical properties. These are used in gears, cams, bearings and seals.NanoZrO2 particles are competent in declining friction co-efficient and wear rate of epoxy composites sliding against steel. The wear resistance of epoxy resin is increased with increasing content of Nano ZrO2.The wear defiant epoxy composite which is filled with ZrO2 particles changed well to the oppose face and its transport film is thin and uniform.Nano-ZrO2 filled epoxy composites show symbols Of mild abrasive wear due to the hard ceramic particles. The main wear mechanism of composites changes from the severe abrasive wear to mild abrasive wear. Mechanical properties are determined by static three point bending.

M.G.Yilmaz, H.Unal, A.Mimaroglu(24) studied the strength and erosive behavior of CaCO3/glass fiber reinforced polyester compound. Here the potency and erosive distinctiveness of CaCO3 filled unsaturated glass fiber/polyester (GFR/UPR) composite are evaluated. Samples of UPR with 40,50,60 weight% contented of CaCO3 and dissimilar CaCO3particle sizes of 1,2,3 and 10 micron are prepared and tested under tensile loading.

Unsaturated polyester is one of the most important thermoset resins in use in application due to handling, molding characteristics and curing properties.CaCO3, glass fiber & carbon block are added into the polymers to improve their stiffness, modulus & reduce their cost. Polymer composites are used in engineering applications that is gears, pumps, impellers where the components undergo erosive wear. The erosive wear of composites are influenced by mechanical properties, fiber content, eroding particle size, impingement angle and velocities. Materials used for experiments are unsaturated polyester resin (UPR), fiberglass (FG), CaCO3 powder and inhibitor. Due to higher percentage of CaCO3 the tensile strength, hardness are high and percentage of elongation at break is less. The maximum erosive wear rate is observed at 90 degree impingement angle. The SEM microscope for UPR/GFR/CaCO3 composites shows the brittle behavior ad cracking mechanism under erosive condition.

N.Mohan, C.R.Mahesha, B.M.Rajaprakash(25) studied the erosive wear behavior of WC filled glass epoxy composites. The effect of tungsten carbide powders on erosive wear behavior I glass fabric epoxy composites is analyzed and the vacuum assisted resin infusion technique is used to fabricate the composite specimen. The result of different impact velocities (40,80m/s) and impact angles are used and the results of erosive wear fatalities impact angle and impact velocities and erosive rate of filled and unfilled glass-epoxy composites are analyzed. The fiber reinforced plastic composites parts such as helicopter rotor blades, high speed vehicle, water turbines, aircraft engine blades, pump impeller blades, aircraft operating in desert environments, missile components. Canopies, radomes and wind screens include purpose in automotive and airspace sectors. The filler materials used are graphite, molybdenum-disulfide, tungsten carbide and silicon carbide. They possesses lf-lubrication properties. Composites fabrication consist of three parts

1. The mixing of epoxy resin and filler using a mechanical stirrer

2. Mixing of the curing agent with the filled epoxy resin

3. Fabrication of composites

The incorporation of WC in G.E composites increases erosion resistance. The effect of impingement particles on erosive wear of every composites exhibits brittle erosive wear actions with utmost erosion speed of 90degree impingement angle.SEM studies of worn surface support the participation of wear mechanisms such as filament breakage, fiber cutting & eroded matrix.

R. SatheshRaja, K.Manisekar, V.Manikandan(26) studied the effect of fly ash filler size on mechanical properties of polymer matrix composite. Particulate composite materials are consisting of polymer resin as matrix and particle as reinforcement. Fly ash is used as filler material in epoxy polymer to manufacture particulate resistant polymer composites. Using simple mold arrangement epoxy resin is varied with 10 wt. % fly ash of dissimilar size. Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS)are utilized to characterize the fly ash. Particulate reinforced polymer (PRP) composite resources are worn in industrialized purpose to have unique physical and mechanical properties. An example of PRP is automobile tire. The experimental materials are poly-iodide, a thermosetting polymer formed from the reaction of an epoxide resin with polyamide hardener.SEM is the most widely used techniques for the chemical and physical characterization of fly ash. The three ordinary method of procedure in SEM scrutiny are backscattered electron imaging (BSE), secondary electron imaging (SEI) &EDS. The remainder beginning the thermal power plant is utilized because filler material in the polymer matrix composites materials. Mechanical testing such as hardness and impact test shows decrease in fly ash filler size increase the hardness.

CHAPTER – III

EXPERIMENTAL METHODOLOGY

3. EXPERIMENTAL METHODOLOGY

3.1MATERIALS USED

The materials used are Iron-oxide powder and epoxy resin to fabricate the composite.

3.1.1 Iron oxide powder

Iron oxide or ferric oxide is the inorganic compound with the formula Fe_2O_3. It is one of the three main oxides of iron, the other two being iron oxide (FeO), which is rare, and iron oxide (Fe_3O_4), which also occurs naturally as the mineral magnetite. As the mineral known as hematite, Fe_2O_3 is the main source of iron for the steel industry. Fe_2O_3 is ferromagnetic, dark red, and readily attacked by acids. Iron oxide is often called rust, and to some extent this label is useful, because rust shares several properties and has a similar composition. To a chemist, rust is considered an ill-defined material, described as *hydrated* ferric oxide.

3.1.2 Epoxy Resin

These are the most commonly used thermoset plastics in polymer matrix composite. These have good adhesion, chemical, environment properties. Filler materials are the inert materials which are used in composites to reduce material cost to improve mechanical properties to some extent.

3.2 Preparation of Samples

The samples were prepared by hand mixing and poured in mold of desired shapes.

3.2.1 Mixing-

Weight percentages of Iron powder and resin, (10%, 20%, and 30%) and (90%, 80%, 70%) were taken respectively. These composition was mixed thoroughly to acquire a homogenous mixture. This composition of iron powder along with resin powder was kept in small size plastic cup. Then harder was taken by an amount $1/10^{th}$ of epoxy resin, and mixed.12,1 inch plastic pipe were cut ,4 for each composition. Lower surface were sealed. Then the mixture were poured inside the pipes. Hence in this way four samples of this composition were made. All the samples were dehydrated in open atmosphere for 2 days. Then the pipes were cut to make the samples.

3.3 Property evaluation

3.3.1 Density

The sample of each composition were weighed in air and immersing in water. The difference is divided by volume is the density.

DENSITY=(M1-M2)/VOLUME*100

Where, M1 and M2 are the mass of dry and wet sample respectively

3.3.2 Hardness

Vickers hardness tester (LECOLM 248AT) as shown in Figure, was used to measure the hardness of all the samples using 300 gf Load. Each data point is the average of five readings.

Figure 3.3 (a) ; Micro Indentation Hardness Tester.

3.3.3 Compressive Strength

In order to measure the compressive strength gauge length and diameter of the specimens were measured individually by the aid of Vernier caliper. The tests were carried out with a crosshead speed of 1mm/min.

Figure 3.3 (b) ; INSTRON Compressive Testing Machine.

3.3.4 Water Absorption

The cylindrical samples were tested for water absorption according to ASTM C642 standard. Now the samples of different composition were immersed in a beaker filled with sea water. After each weak weight of samples were measured. The amount of water absorbed was calculated by weight change.

3.3.5 Dielectric behavior

A dielectric is an electrical insulator that may be polarized by imposition of an electric field. When a dielectric is positioned in an electric field, electric charges do not flow through the material, as in a conductor but only slightly move from their standard stability positions making dielectric polarization. Because of dielectric split, positive charges are relocate beside the field and negative charges move in the opposite direction. This makes an inside electric field which partially compensates the exterior field within the dielectric. If a dielectric is composed of feebly bonded molecules those molecules not only develop polarized, but also reorient so that their equilibrium alignment aligns to the playing field.

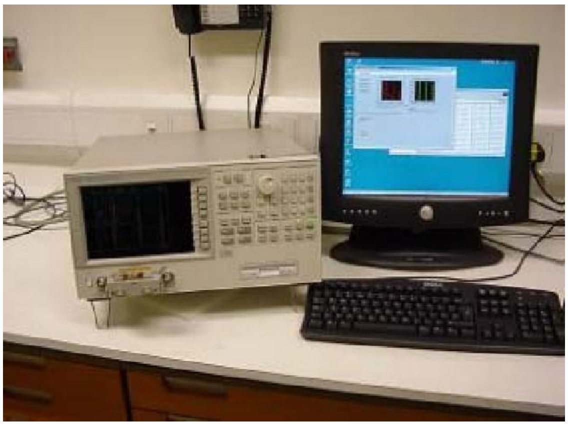


Figure 3.3 (c) ; Dielectric Interface Equipment.

While the expression "insulator" refers to a low degree of electrical conduction, the word "dielectric" is typically worn to explain materials with a high polarizability. The latter is articulated by a integer named the dielectric constant. A universal, however remarkable, instance of a dielectric is the electrically insulating substance among the metallic dishes of a capacitor. The polarization of the dielectric by the practical electric field increases the capacitor's capacitance. The knowledge of dielectric performance is anxious with the storage and dissipation of electric and magnetic force in equipment.

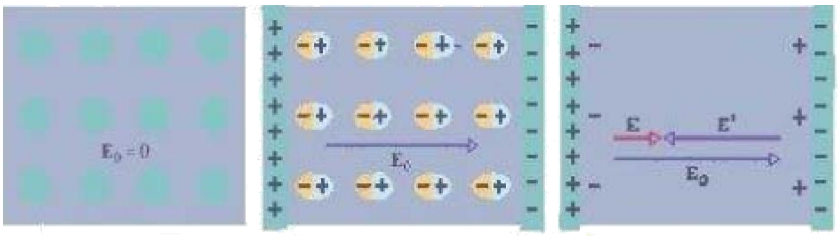


Figure 3.3 (d) ; Polarization of the Dielectric Material between the Capacitor Plates.

3.3.6 Sliding Wear

In this study computerized Pin on disc friction and Wear Test rig as shown in Figure was used to evaluate the wear performance and sliding contact resistance of the polymeric samples. The experiment was carried out with the help of diamond indenter keeping the different track radius 30 mm respectively. Prior to wear, constant normal load of 30N and 60N was applied. The indenter rotates on samples with speeds of 100,200,300 rpm for different time period of 600s. At the end of each test, loss in weight of the samples was noted. Results obtained have been expressed in terms of wear depth, and friction co-efficient.

Figure 3.3 (e) ; Pin On Disc Friction And Wear Test Machine.

3.4 Micro structural Characterization

3.4.1 SEM analysis

In the present study, a JEOL Scanning Electron Microscope (Fig. 3.4) was used for the observation of micro structural changes (pits, cavities, and porosity and wear track etc.) and determination of particle size and morphology of composites. To acquire the improved picture resolution, secondary electron imaging with accelerating voltage of 15 KV was used.

Figure 3.4 (A) ; Scanning Electron Microscopy (JEOL JSM-6480LV).

CHAPTER – IV

RESULTS AND DISCUSSION

4. Result and Discussion

4.1 Composition of Iron Powder

The iron oxide powder is mixed with epoxy resin and ispoured in plastic mold. The ratio of iron powder and epoxy resin are 1:9(composition 1), 2:8(composition 2), 3:7(composition 3), shown in the table 4.1.

Name of sample	Composition of iron powder In percentage
1	10
2	20
3	30

Table 4.1 ; Compositional Analysis of Iron Powder.

4.2Density Measurement

Density of sample was measured by water treatment methods. From the table we can say density increases with increase in iron powder.

Mix Composition (wt. %)	Density(gm/cc)
IP (10%) +ER (90%)	1.619
IP (20%) +ER (80%)	1.73
IP (30%) +ER (70%)	2.019

Table 4.2 ; Density of Dry Sample.

Fig.4.2 shows a relation between densities of dry composite with respect to iron powder composition. It is evident from the graph that the density is increased with the addition of iron powder.

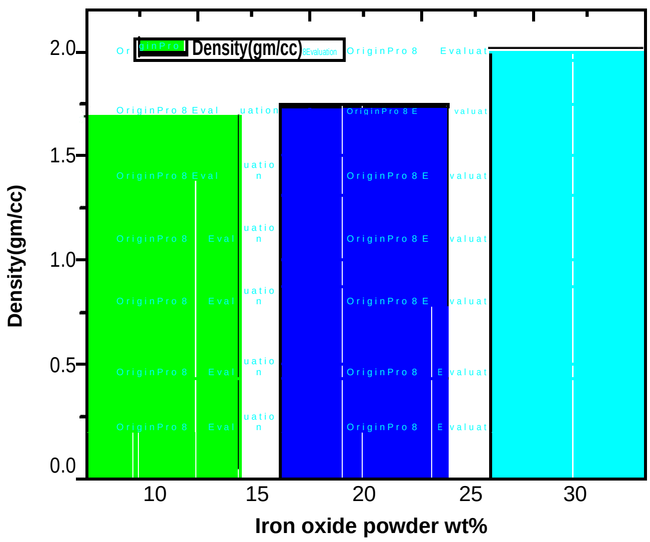


Figure 4.2 ; Variation of Density with Iron Powder.

4.3 Sea Water Treatment

Table 4.3(a) shows the amount of weight change due to water absorption in different composition of the composites. Water absorption is the difference between the weight of wet sample and the weight of dry sample. After water treatment the density of all composition can be calculated by formula weight difference/volume.

Time(week)	Weight change of composition1(gm)	Weight change of composition2(gm)	Weight change of composition3(gm)
1 day	3.312	3.358	3.404
1st week	3.3467	3.3607	3.428

2nd week	3.3678	3.392	3.474
3rd week	3.3856	3.405	3.495
4th week	3.392	3.428	3.507

Table 4.3(A) ; Weight of Different Samples after Water Treatment.

Time(week)	Water absorption of composition1	Water absorption of composition2	Water absorption of composition3
1	.0347	.0027	.024
2	.0211	.0313	.046
3	.0178	.013	.021
4	.0064	.023	.012

Table 4.3(B) ; Water Absorption of Different Composition after Water Treatment.

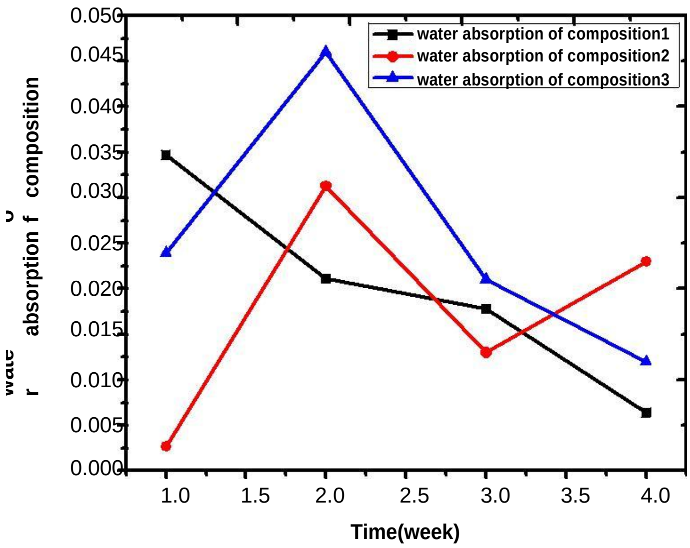


Figure 4.3 (a) ; Water Absorption with Time.

Time(week)	Density change of	Density change of	Density change of

	composition1	composition2	composition3
1	.0141	.0012	.0121
2	.0227	.0151	.0327
3	.0301	.021	.0424
4	.0327	.0312	.0481

Table4.3(C) ; Density of Different Composition after Water Treatment.

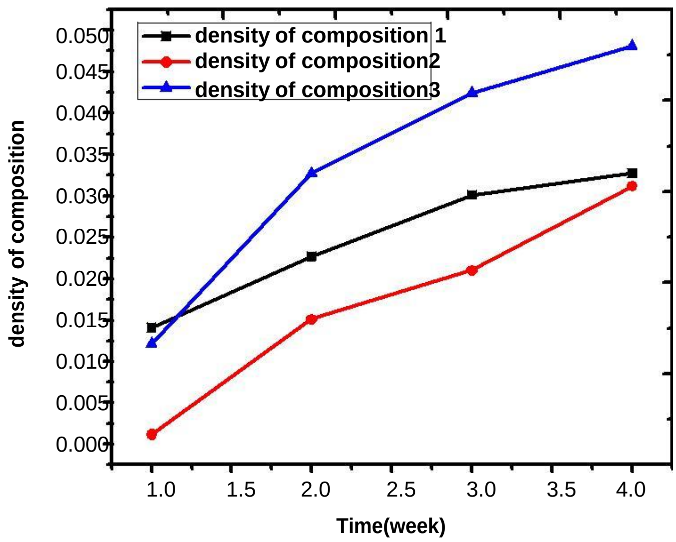


Figure 4.3 (b) ; Variation of Density with Time.

4.4 Hardness Measurement

Hardness values of all the composites of different compositions were measured using LECO, LM 248AT Vickers hardness tester. The hardness was measured at different position of samples of each composition. The obtained values of hardness are given in the table 4.3.

Sample-1

Reading	Diameter(D1) In micrometer	Diameter(D2) in micrometer	Hardness value(Vicker, HV)

1	171.1	165.5	19.6
2	156.3	175.4	20.2
3	175.4	159.8	18.7
4	168.8	159.8	20.6

Table 4.4(a) ; Hardness Value Of Composition 1.

Reading	Diameter(D1) in micrometer	Diameter(D2) in micrometer	Hardness value(Vicker, HV)
1	163.9	160.6	21.1
2	158.3	162.7	21.6
3	167.4	170.1	19.5
4	161.2	155.8	22.1

Table 4.4(b) ; Hardness Value Of Composition 2.

Reading	Diameter(D1) In micrometer	Diameter(D2) In micrometer	Hardness value(Vicker, HV)
1	176.2	175.6	21.2
2	170.6	169.0	21.4
3	176.4	181.0	22.4
4	178.9	182.9	22.6

Table 4.4(c) ; Hardness Value of Composition 3.

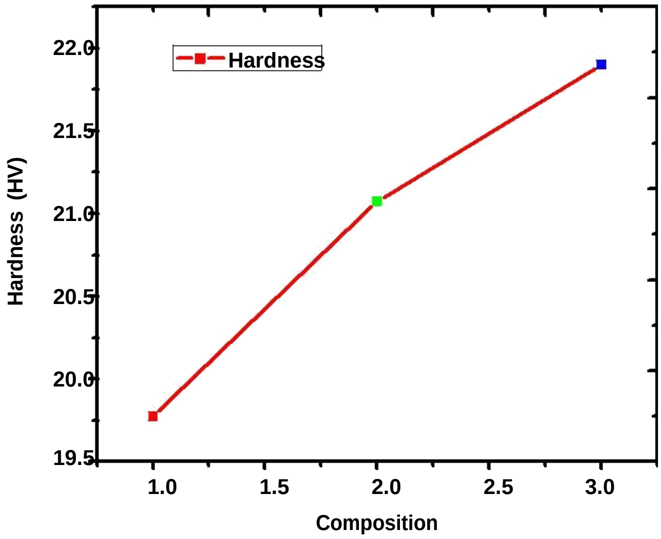


Figure 4.4 ; Variation of Hardness with Composition.

4.5 Compressive Strength measurement

The compressive strength measurement of the cylindrical samples was done by INSTRON. Test was conducted on the one sample of each composition and the standard value of all are to be evaluated. The strength values of different compositions in dry state is shown in table 4.5.

Specimen no.	Stress at maximum(Mpa)	% Strain at maximum (%)	Compressive strength(mpa)
1	224.6	72.31	112.7
Mean	224.6	72.31	112.7

Table 4.5 (a) ; Compressive Strength Value of Composition 1.

Specimen no.	Stress at maximum(Mpa)	% Strain at maximum (%)	Compressive strength(mpa)

1	112.7	6.842	120
Mean	112.7	6.842	120

Table 4.5 (b) ; Compressive Strength Value of Composition 2.

Specimen no.	**Stress at maximum(Mpa)**	**% Strain at maximum (%)**	**Compressive strength(mpa)**
1	120.0	6.428	224.6
Mean	120.0	6.428	224.6

Table 4.5 (c) ; Compressive Strength Value of Composition 3.

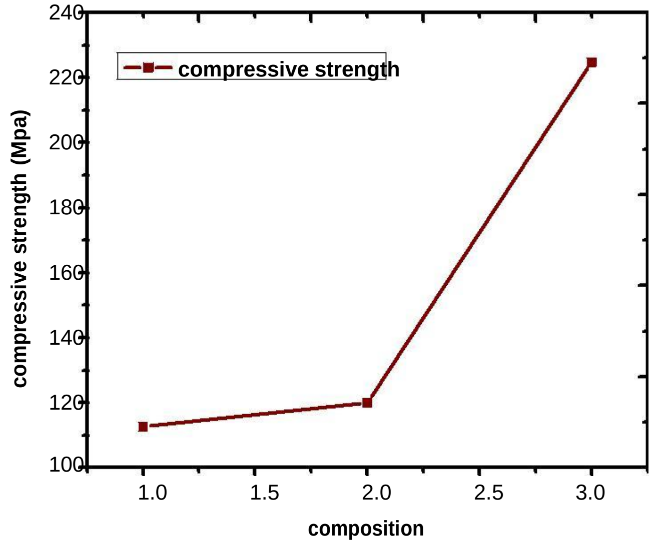


Figure 4.5 ; Compressive Strength of Composites.

4.6 Dielectrics Study

In dielectric examination, the top and bottom face of the sample is coated with silver paint for the purpose of conductance; themodel is positioned among the two Al parallel electrodes. A sinusoidal signal is applied, creating a discontinuous electric field. This electric field create polarization in the sample ,which oscillates at the identical frequency as of the practical electric field, but has a phase angle shift δ .This phase angle shift is measured by comparing the applied voltage to the calculated current , which is divided in to capacitive and conductive components

The dielectric constant, dielectric loss, conductivity and resistivity are determined as follows.

Dielectric constant $K = C'/C$.

Where C'(pF) is measured capacitance and C (pF) is calculated using the formulae,

$C = \varepsilon 0. (A/d)$;

A ($m.m^2$) - area of the electrode, d (m. m)-thickness of the sample.

Figure 4.6 gives the variation of dielectric constant, and tangent loss with frequency at room temperature. Figure 4.6(a) shows the variation of tan delta with frequency for different composition of iron powder and epoxy resin (1:9).Figure shows that with increase in iron powder tan delta value increases.

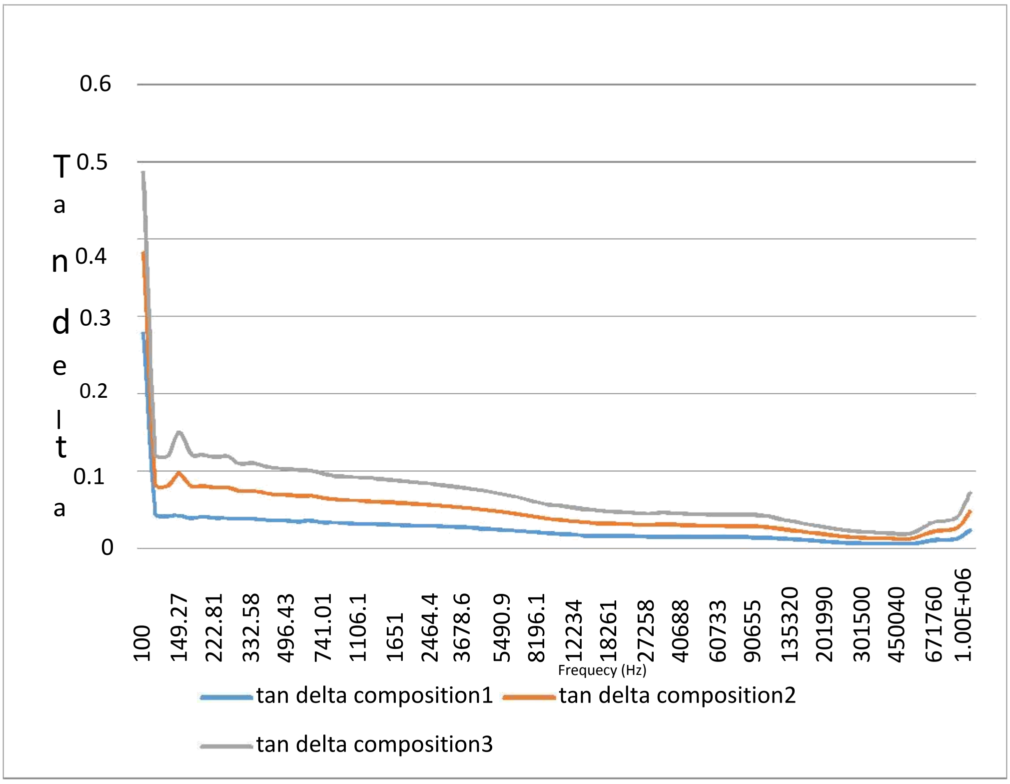


Figure 4.6 (a) ; Frequency Vs Tan Delta of Different Composition.

Figure4.6 (b) shows the variation of capacitor with frequency of all composition of iron powder and epoxy resin.

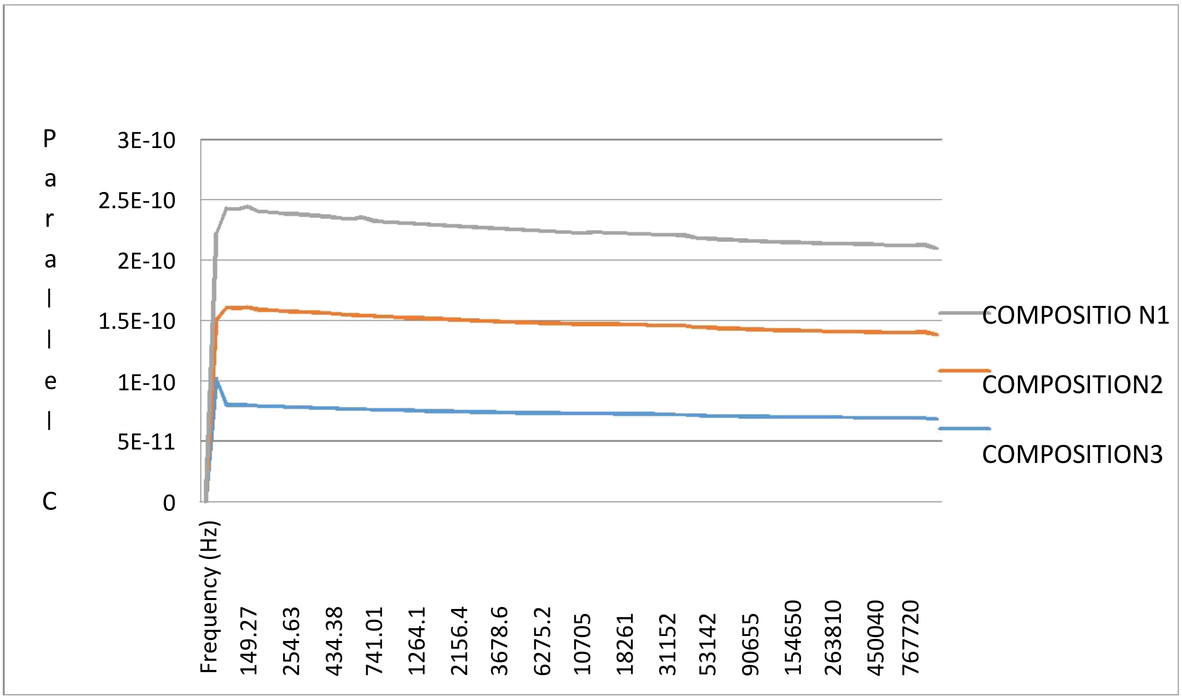


Figure 4.6 (b) ; Variation of Capacitor with Frequency.

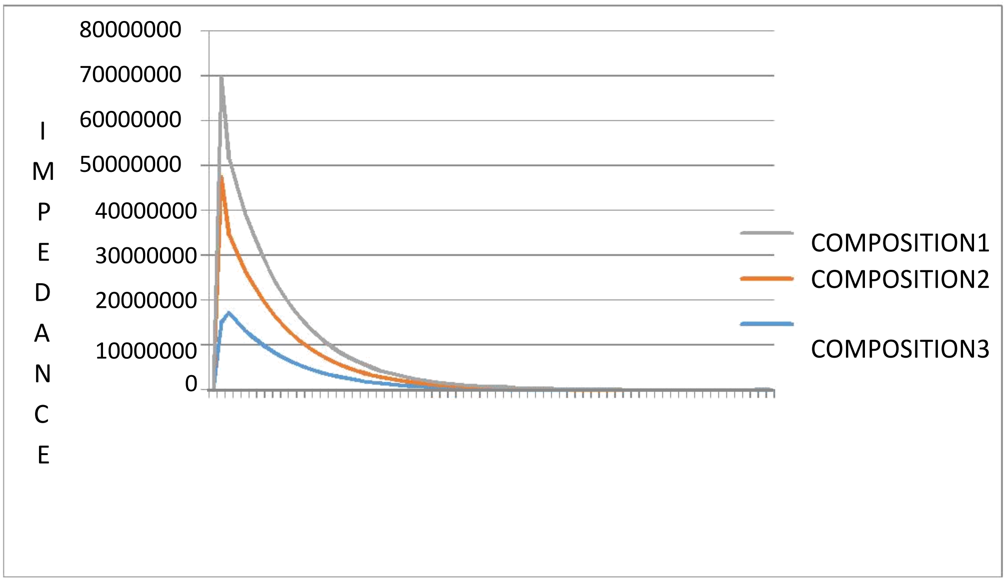


Figure 4.6(c) ; Variation of Impedance with Frequency.

From the above figures, it is clear that the dielectric constant decreases with increasing frequency as it is expected behavior for most of the materials. With frequency tan delta, impedance and capacitance initially increases then continuously decreases. As the frequency increases, charges become more random and start to oscillate out of phase with the applied voltage and will contribute to the alternating current causing decrease in K value. It is also seen that the dielectric constant is in decreasing order.

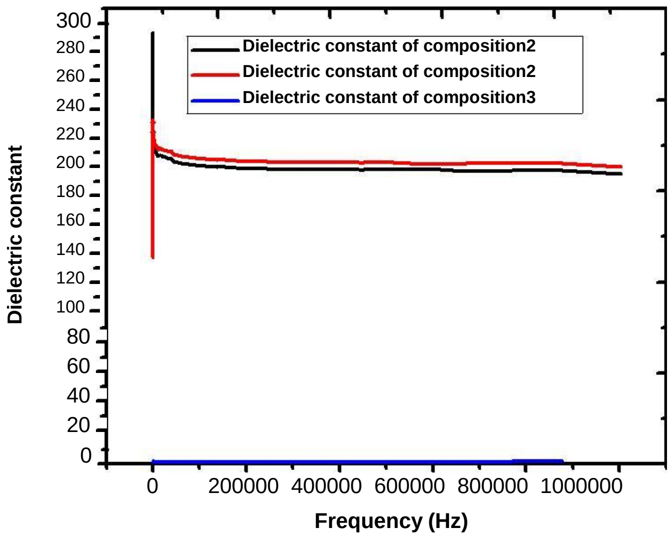


Figure 4.6 (d); Variation of Dielectric Constant with Frequency.

Dielectric study after water treatment

Figure 4.6(e-g)) shows the variation of dielectric constant, tan delta, impedance, capacitance and tangent loss with frequency at room temperature for different composition of the composites. Figure shows that with increase in iron powder tan delta value increases.

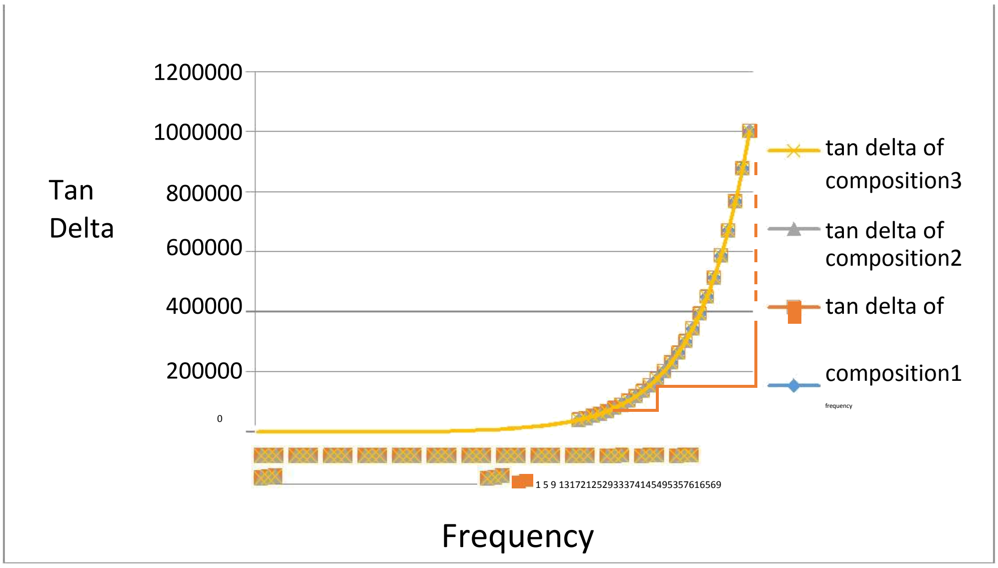


Figure 4.6 (e) ; Variation of Tan Delta with Frequency.

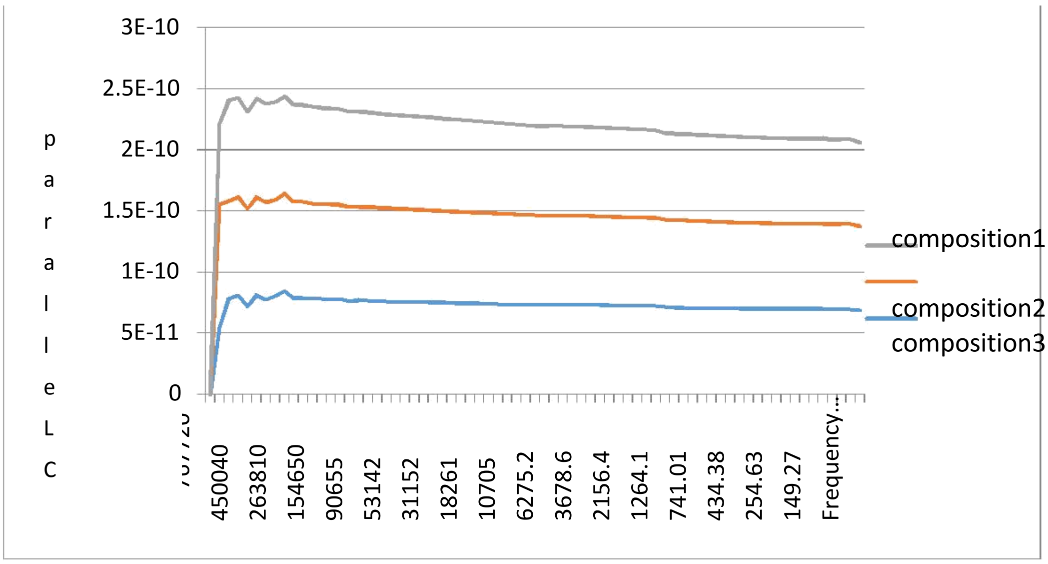


Figure 4.6 (F) ; Variation of Capacitance with Frequency.

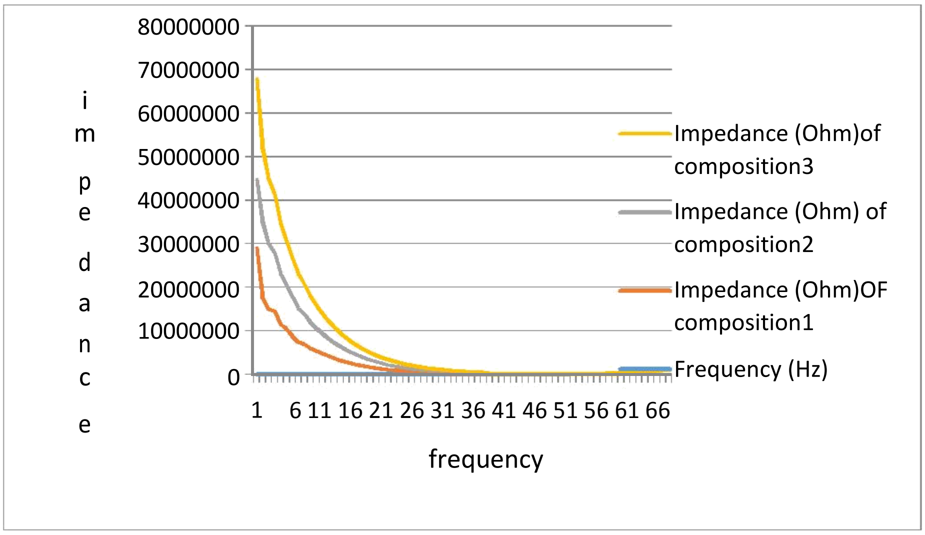


Figure 4.6(g) ; Variation of Impedance with Frequency.

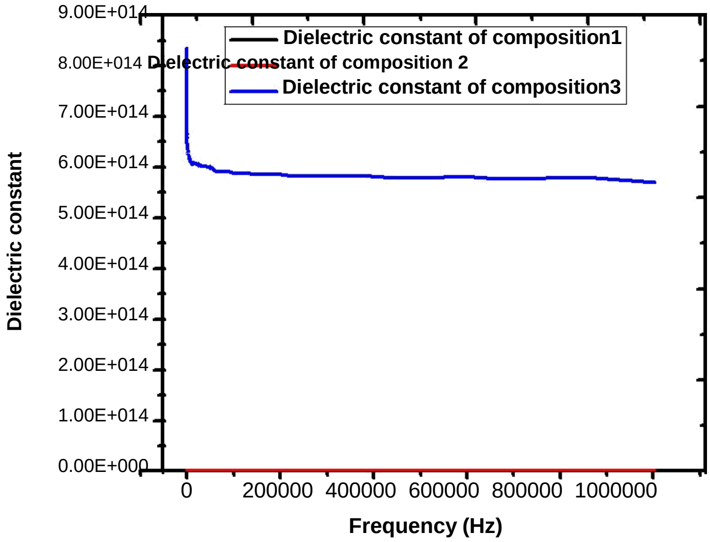


Figure 4.6 (h); Variation of Dielectric Constant with Frequency.

From the figures, it is clear that the dielectric constant decreases with increasing frequency as it is the expected for most of the materials having low dielectric constant. With frequency tan delta, impedance and capacitance all first increases then gradually decreases.

4.7 Evaluation of Wear behavior:

Sliding wear test was carried out at room temperature using pin on disc wear tester. A disc made of hardened chromium steel was used as the counter body against the pin (sample).The test was conducted at 100,200.300 rpm on a track diameter 60mm.The other parameters varied in the experiment were sliding velocity, load and sliding distance. Applied Loads were 30N and 60N.The disc was cleaned with acetone and evenness of disc surface is measured before and after each run. The specimen was held stationary and disc was rotated while a normal force was applied through a lever arm. A computer aided data acquisition system is attached to record wear, frictional force and co-efficient of friction.

Wear characteristics of polymer composites were carried out at different loads of 30N,60N and speed of 100rpm, 200rpm and 300rpm.Graphs 4.7 are showing the variation of wear (micro),frictional force (N) and coefficient of friction of different composition.

Figures show that by applying load 30N and speed 100rpm,the wear rate, frictional force and co-efficient of friction of all three composition. The wear rate is increased with increase in time as well as the frictional force and co-efficient of friction are rising with increase in sliding time.

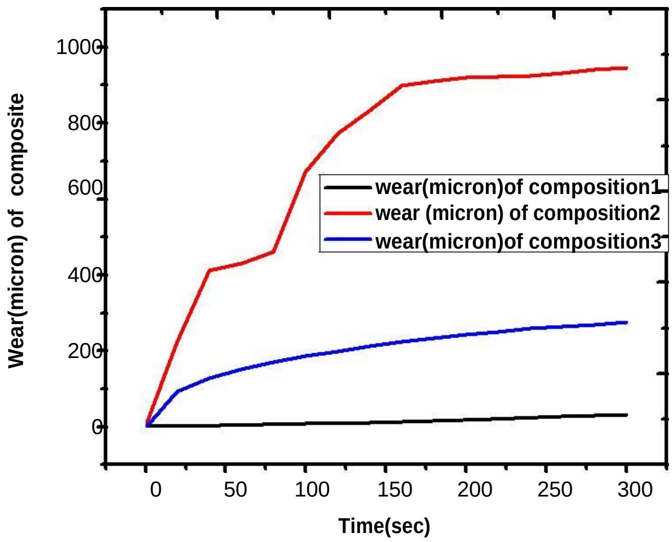


Figure 4.7 (a) ; Wear Characteristics of Different Compositions.

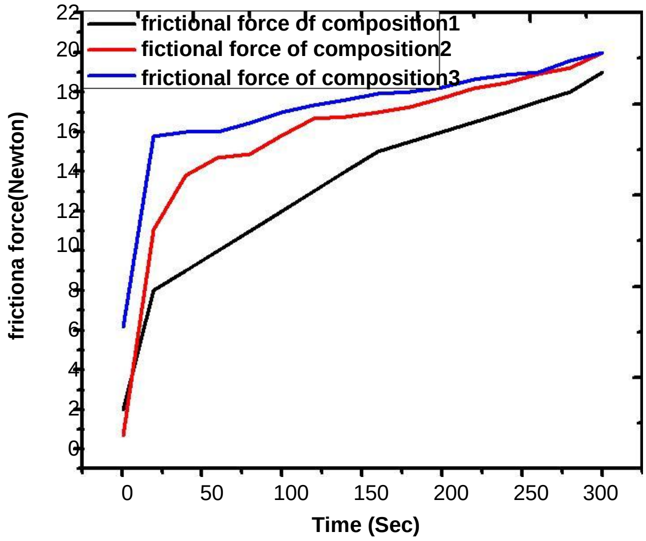


Figure 4.7 (b) ; Frictional Force Characteristics of Different Composition.

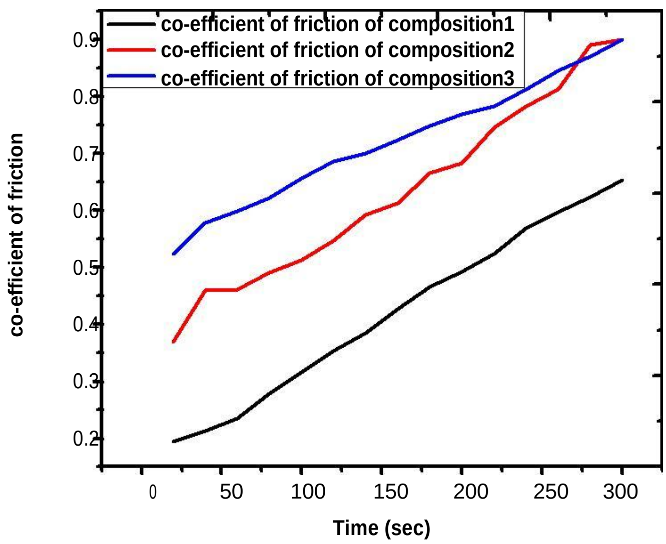


Figure 4.7 (c) ; Co-Efficient of Friction of Different Composition.

Figure (4.7 d-f) shows that by applying load 30N and speed 200rpm,the wear rate, frictional force and co-efficient of friction of all three composition of varying iron powder. The wear rate is increased with increase in time and the frictional force and co-efficient of friction are increasing with increase of time.

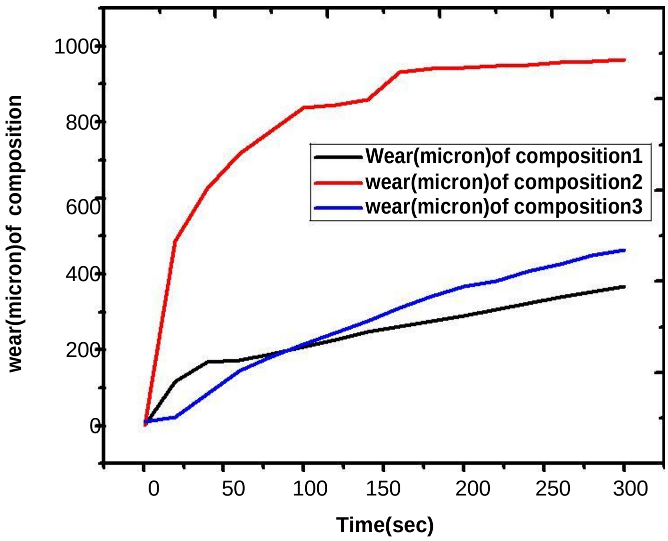


Figure 4.7 (d) ; Wear Characteristics of Different Composition.

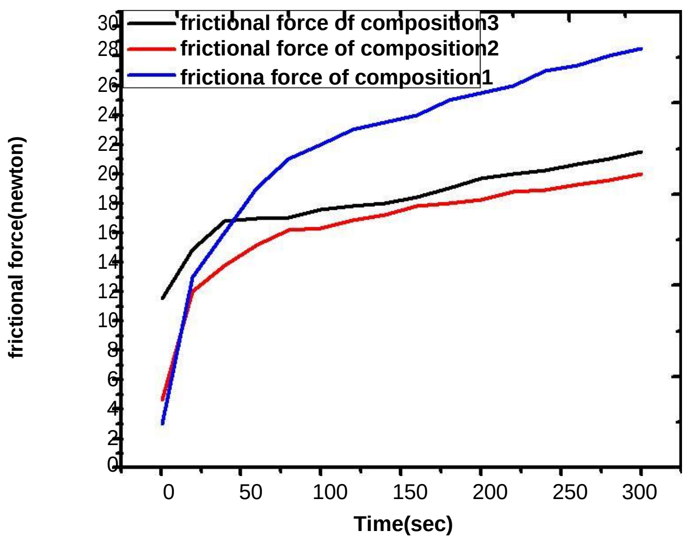


Figure 4.7(e) ; Frictional Force Characteristics of Different Composition.

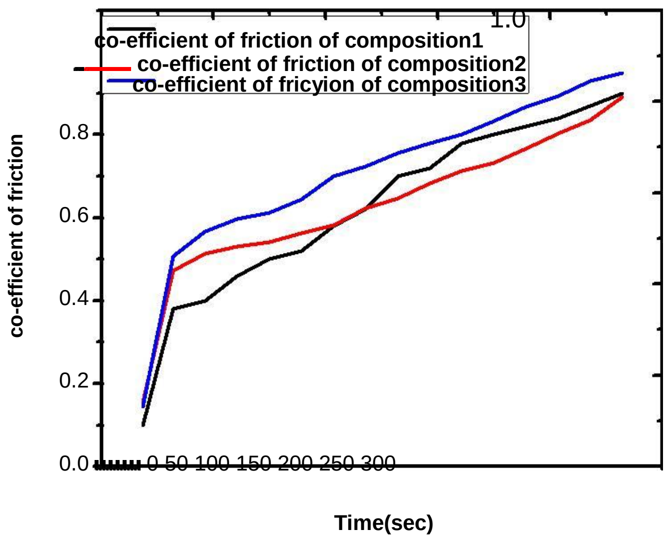


Figure 4.7 (f) ; Co-Efficient of Friction of Different Composition.

Figure 4.7 (g, h, i) shows that by applying load 30N and speed 300rpm, the wear rate, frictional force and co-efficient of friction comparison of all three composition of varying iron powder. The wear rate is increased with increase of time as well as the frictional force and co-efficient of friction are also increasing with increase of time.

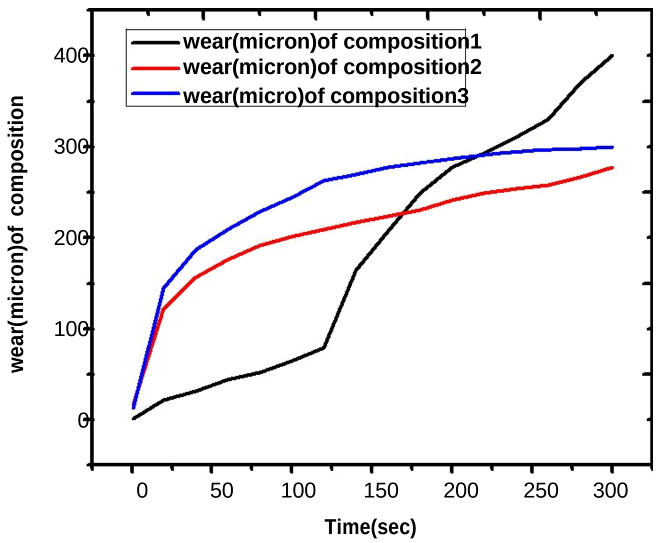


Figure 4.7 (g) ; Wear Characteristics of Different Composition.

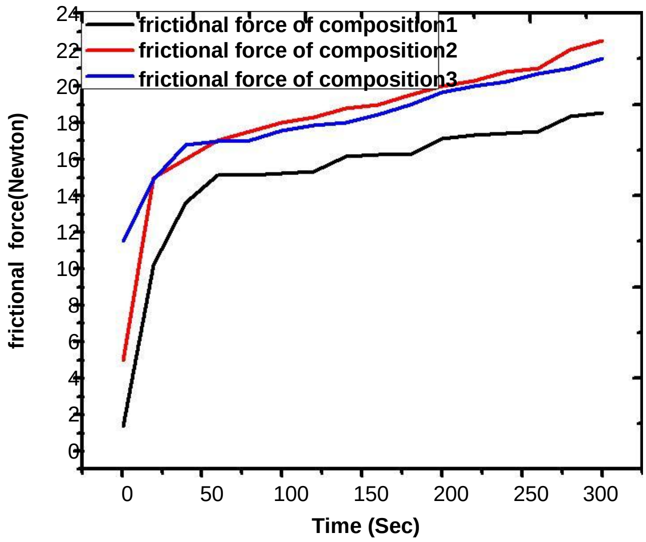


Figure 4.7 (h) ; Frictional Force Characteristics of Different Composition.

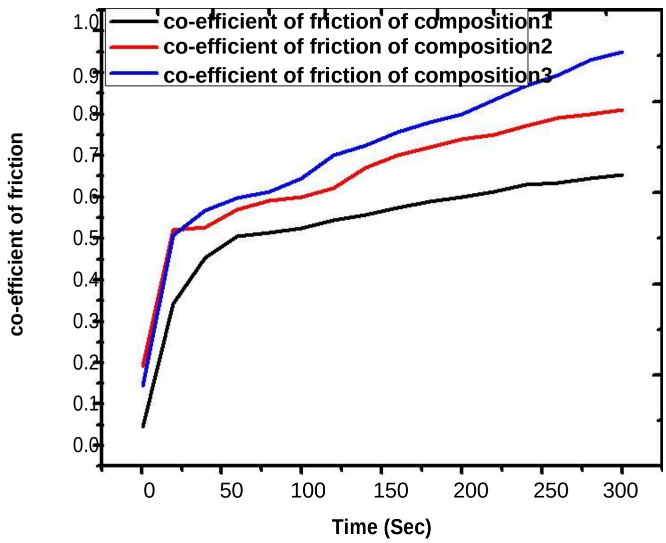


Figure 4.7(i) ; Co-Efficient of Friction of Different Composition.

Figure 4.7 (j, k, l) shows that by applying load 60N and speed 100rpm,the wear rate ,frictional force and co-efficient of friction comparison of all three composition of varying iron powder. The wear rate is increased with increase of time as well as the frictional force and co-efficient of friction are also increasing with increase of time. The wear rate, frictional force and co-efficient of friction increases with increase in iron powder content.

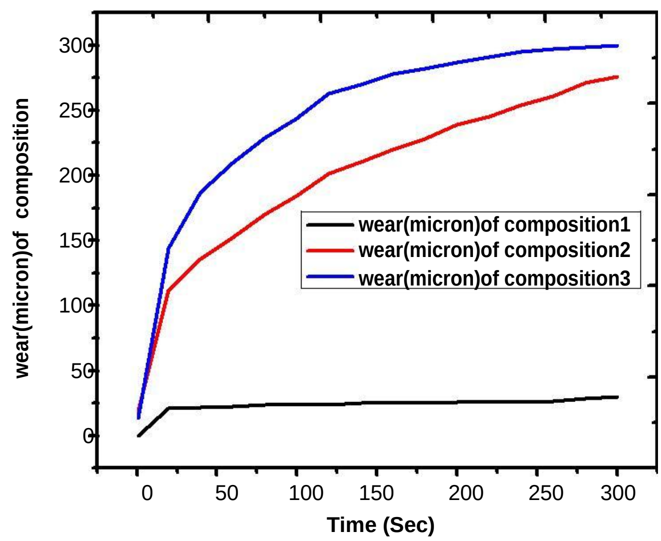


Figure 4.7 (j); Wear Characteristics of Different Composition.

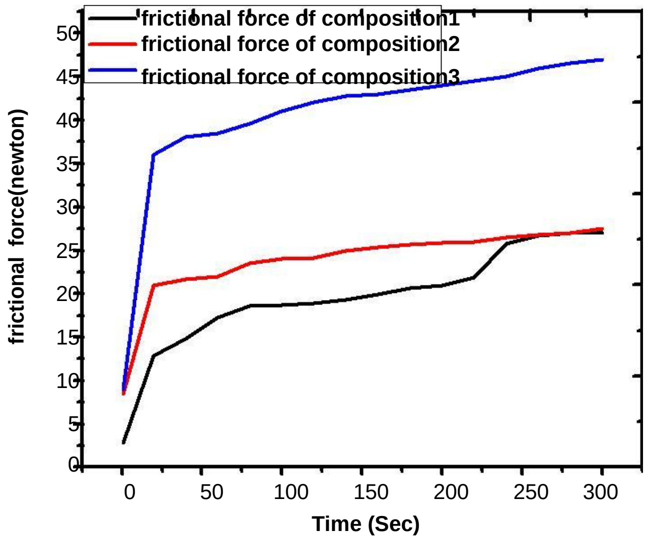


Figure 4.7 (k); Frictional Force Characteristics of Different Composition.

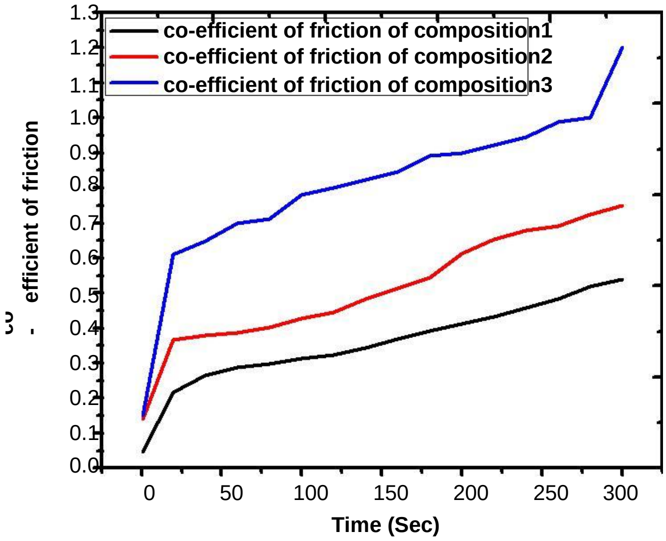


Figure 4.7 (l) ; Co-Efficient of Friction of Different Composition.

Figure (4.7 m, n, o) shows that by applying load 60N and speed 200rpm, the wear rate, frictional force and co-efficient of friction comparison of all three composition. The wear rate is increased with increase of time as well as the frictional force and co-efficient of friction are also increasing with increase in time.

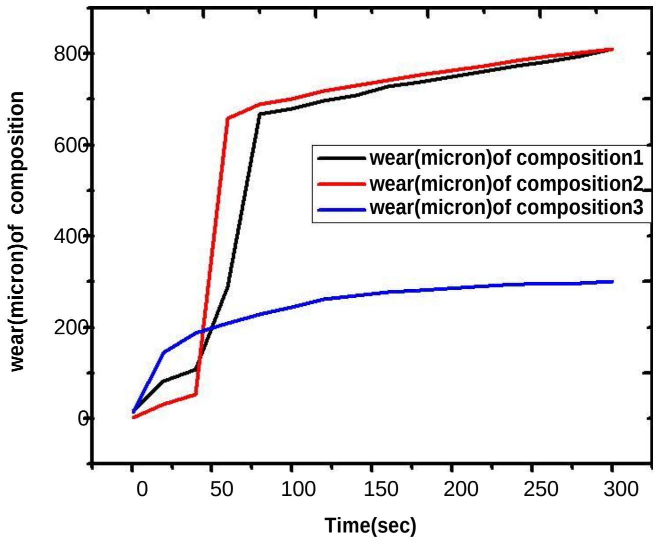


Figure 4.7 (m) ; Wear Characteristics of Different Composition

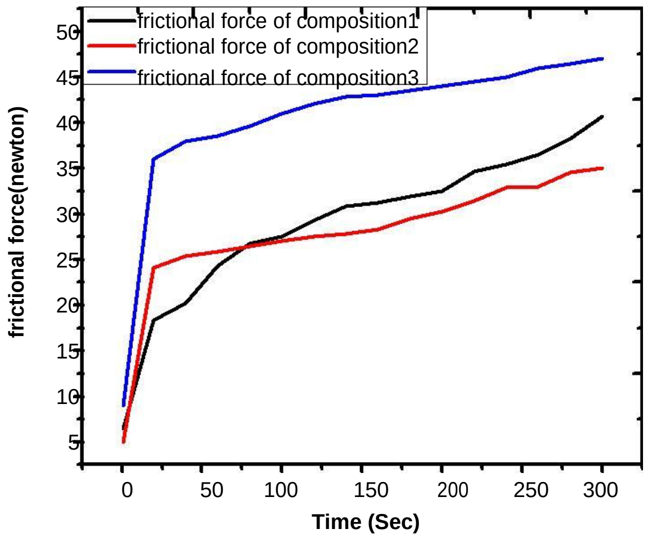


Figure 4.7 (n) ; Frictional Force Characteristics of Different Composition.

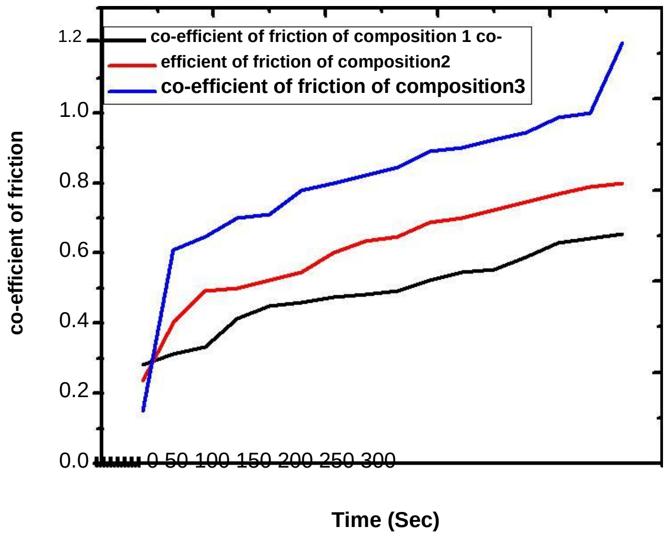


Figure 4.7 (o) ; Co-Efficient of Friction of Different Composition.

Figure 4.7 (p, q, r) shows that by applying load 60N and speed 300rpm, the wear rate, frictional force and co-efficient of friction for all three compositions. The wear rate is increased with increase in time as well as the frictional force and co-efficient of friction are also increasing with increase of time.

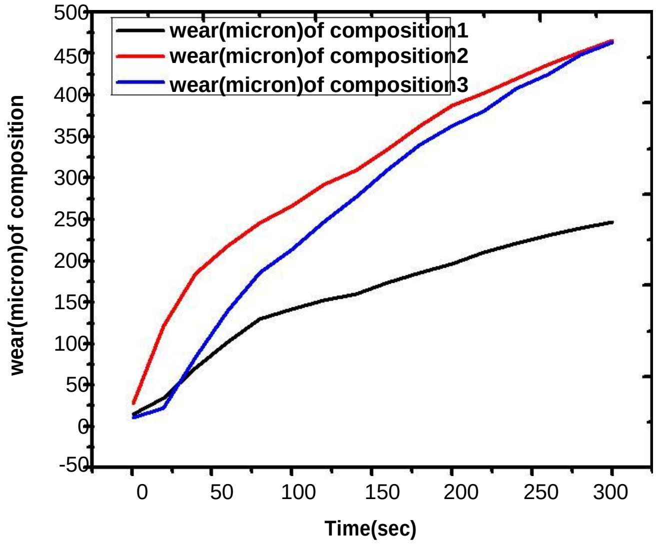


Figure 4.7 (p) ; Wear Characteristics of Different Composition.

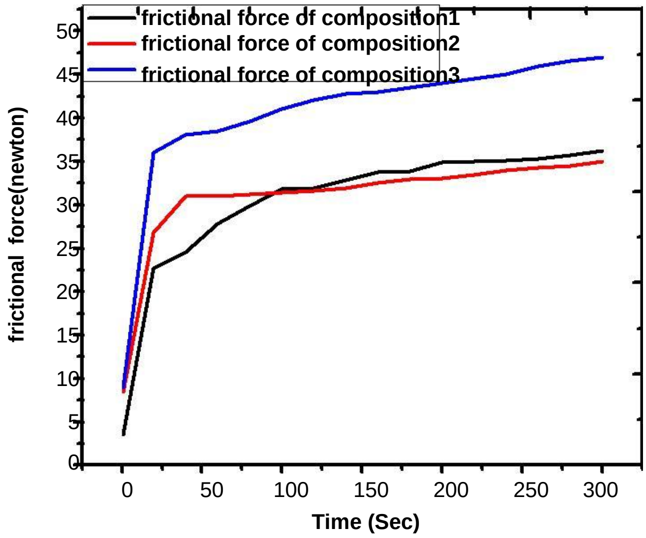


Figure 4.7 (q) ; Frictional Force Characteristics of Different Composition.

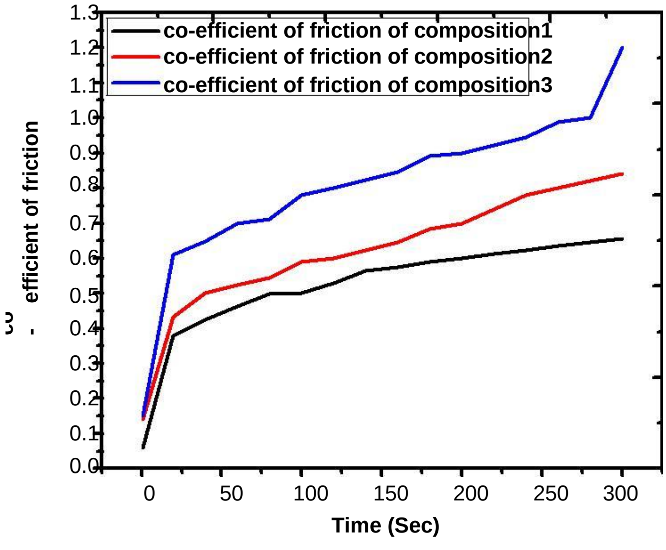


Figure 4.7(r); co-efficient of friction of different composition.

4.8 Evaluation of wear behavior after water treatment

The samples were dried. Wear characteristics of composites were carried out at 50N load and speed of 200 rpm. Graphs 4.8 (a, b, c) are showing the wear (micro),frictional force (N) and coefficient of friction characteristics respectively. It is seen from the graphs that wear rate is increasing with time.Composition2 has higher wear rate than composition1 and composition3.

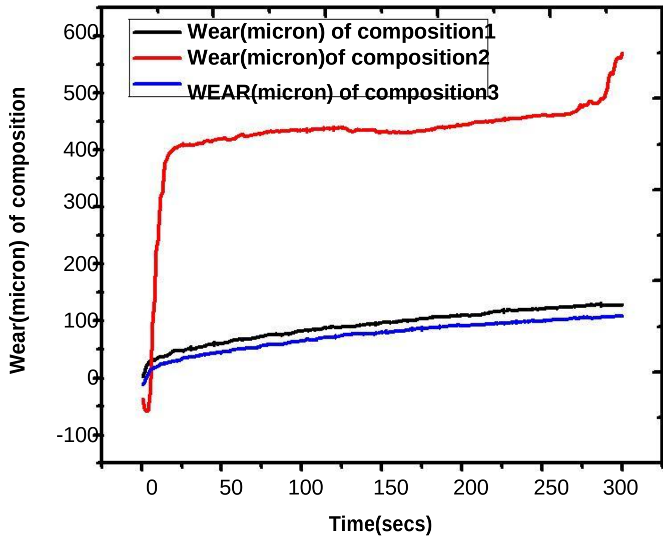


Figure 4.8 (a) ; Wear Characteristics of Different Composition

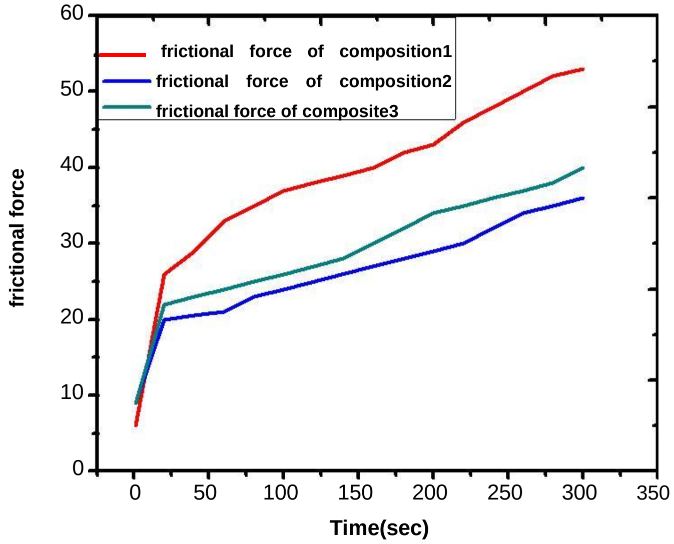


Figure 4.8 (b) ; Frictional Force Characteristics of Different Composition.

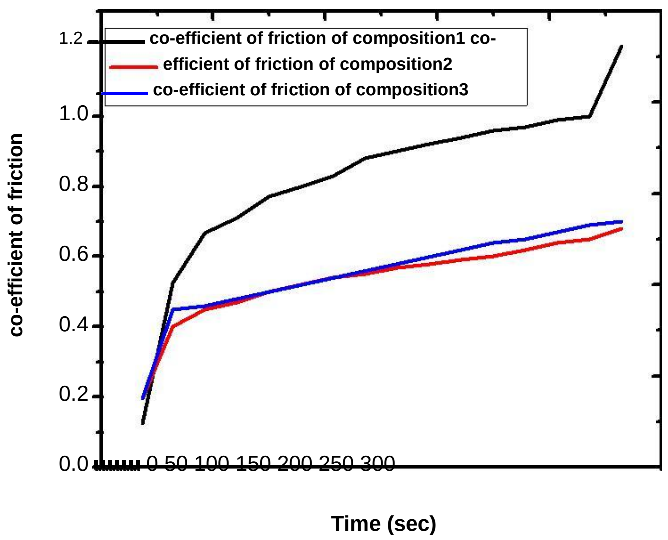
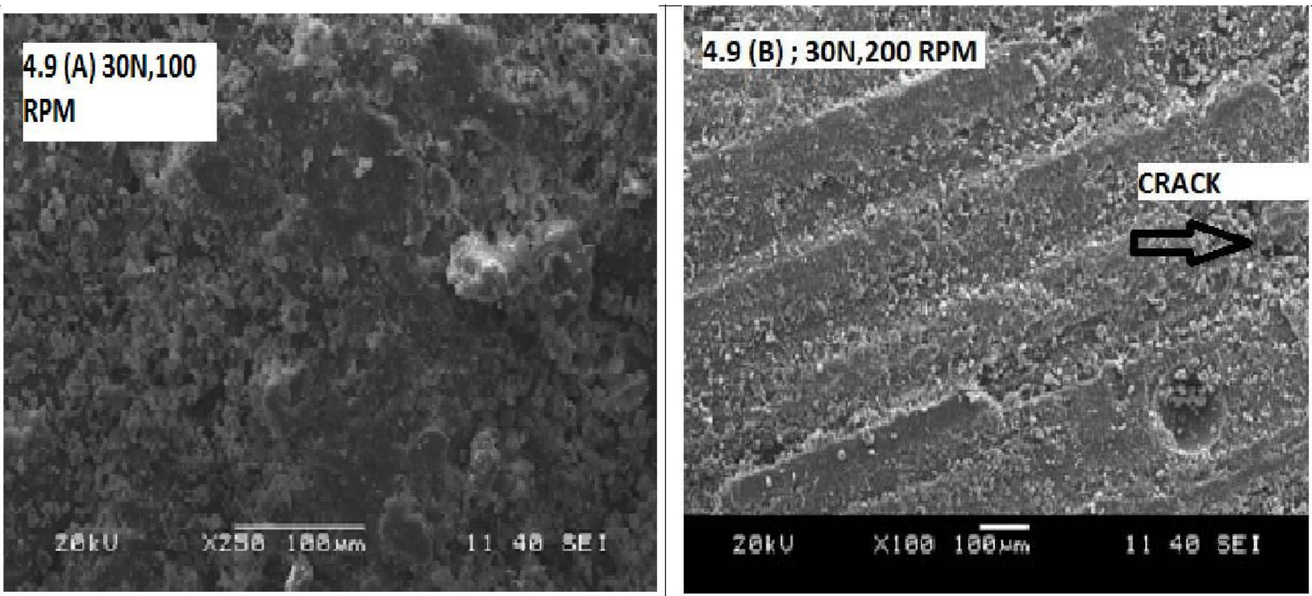
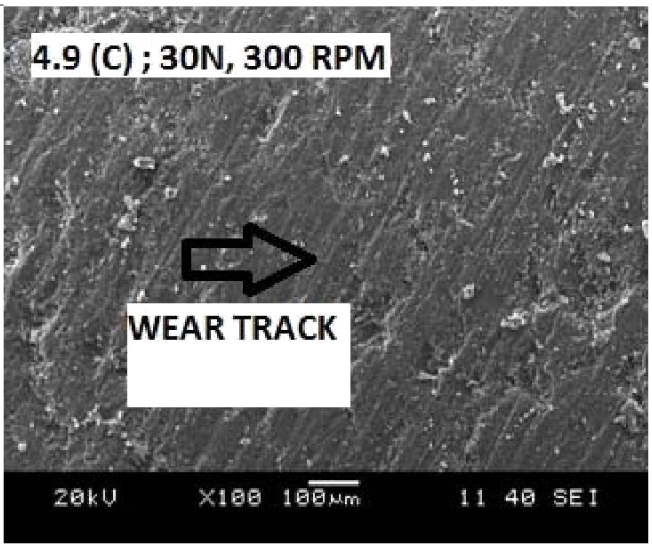


Figure 4.8 (c) ; Co-efficient of Friction of Different Composition.

4.9 SEM Analysis:

Wear behavior can be classified under ductile and brittle categories. Thermoplastic matrix composites exhibit ductile behavior, while thermosetting matrix composites show brittle erosion. The surface morphology of the composites was examined using a scanning electron microscope (SEM).The basic wear mechanism in thermoset composites are matrix micro-cracking, matrix deboning, material removal, plastic flow of material etc. Surface and subsurface degradation of the iron oxide powder reinforced polymer composite after sliding wear test performed on pin on disc wear tester with different loads and different velocities, was analyzed by SEM.SEM imaging illustrate the elimination of resin substance from impacted surface. In the image the load and velocity used are indicated. Under all set of sliding condition debris formation took place. The largest wear tracks surrounded by high amount of wear debris, plastic flow of epoxy.





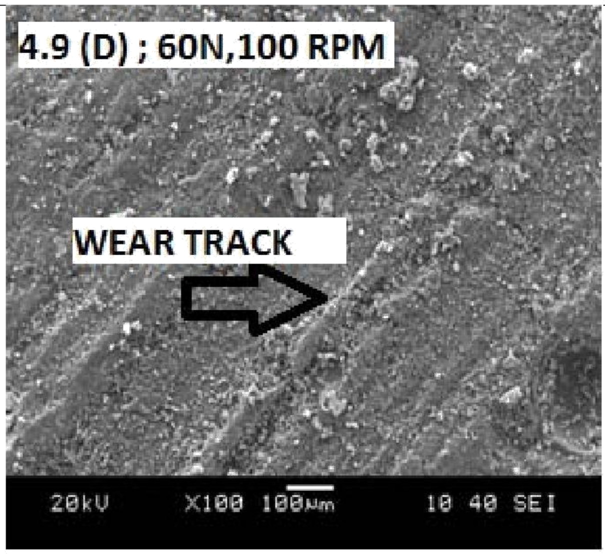


Figure 4.9 (A,B,C,D) ; SEM Microstructure of Wear Track

From the above SEM microstructure it is found that, with decrease epoxy resin and increase iron oxide powder it is seen that the interface bonding becomes better and less amount of crack at the interface. In figure micro crack are initiated. Also, the figures show comparatively more damage to surface which is due to increase in load and velocity.

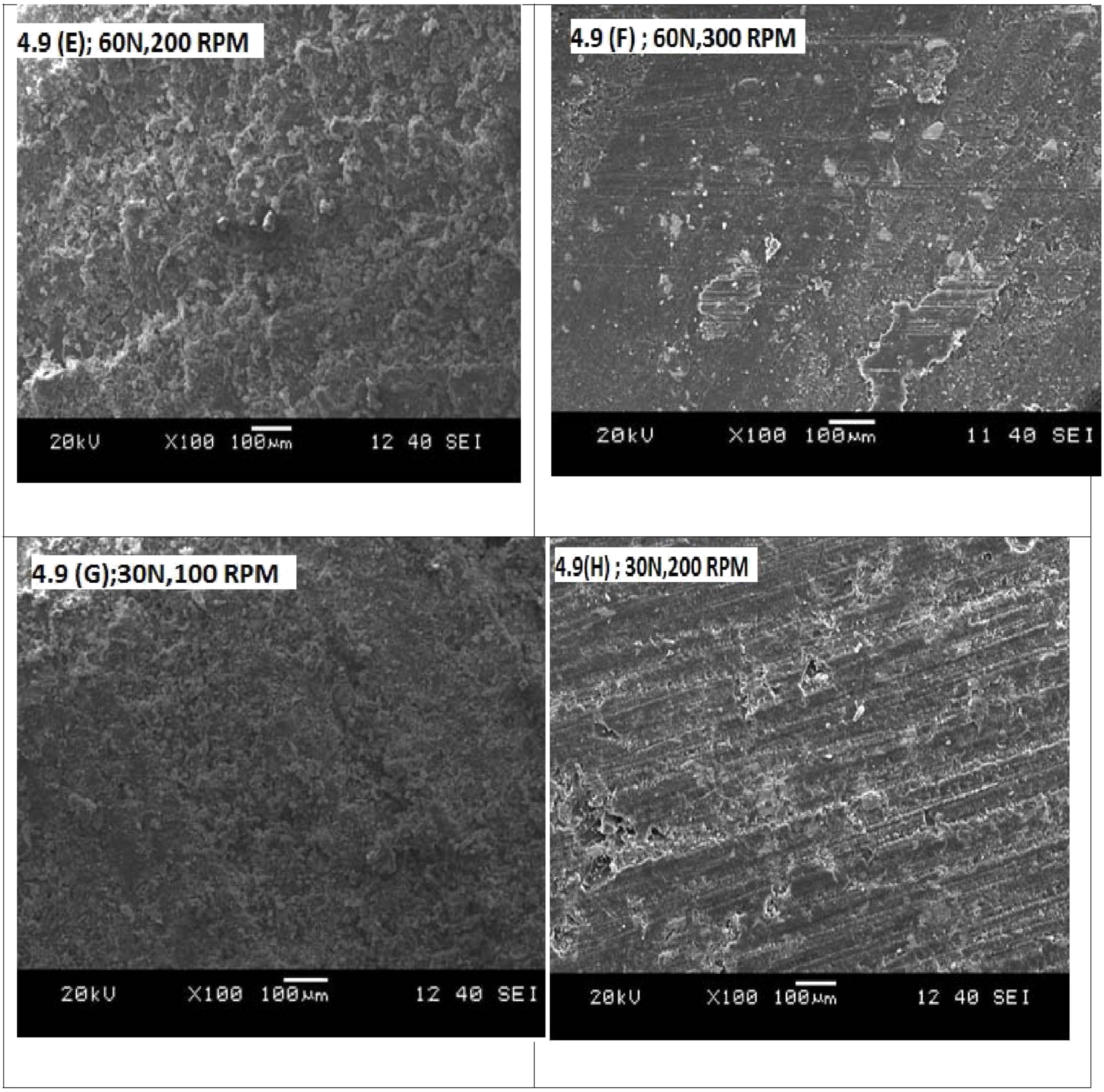


Figure 4.9 (E,F,G,H) ; SEM Microstructure of Worn surface.

Above figures show that, the SEM micrograph of wear track along the sliding direction. The images show that the wear mechanism is basically formation of crack, delamination etc. Layer formation of the rubbed particles takes place in the above figure. Some particles are deboned from the matrix.

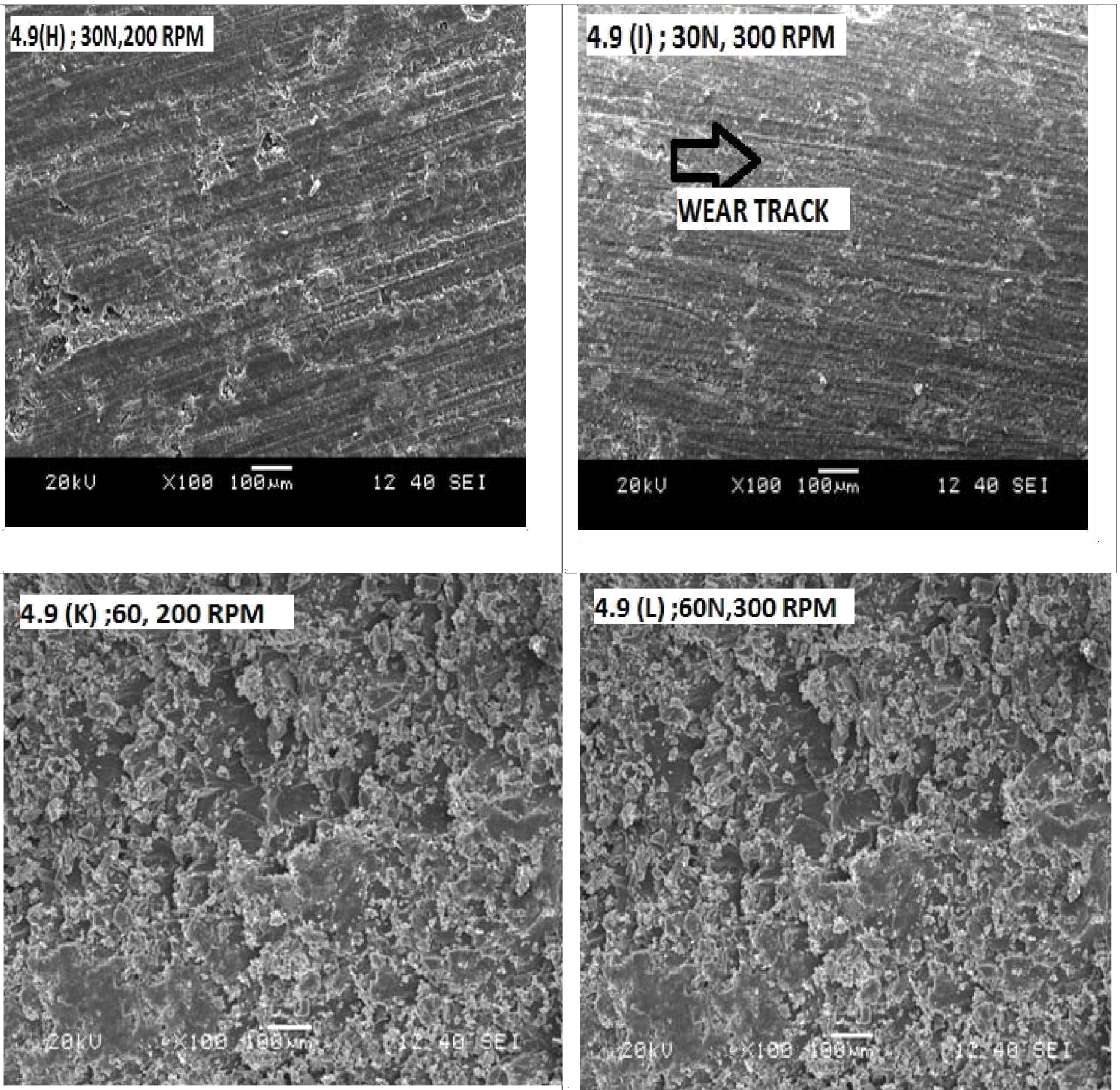


Figure 4.9 (I,J,K,L) ; SEM Microstructure of Worn surface.

Wear tracks are formed during multi-pass wear owing to micro-cutting. It is apparent from the microstructure that initially micro-cracks are formed during sliding. Plastic flow of epoxy is also seen.

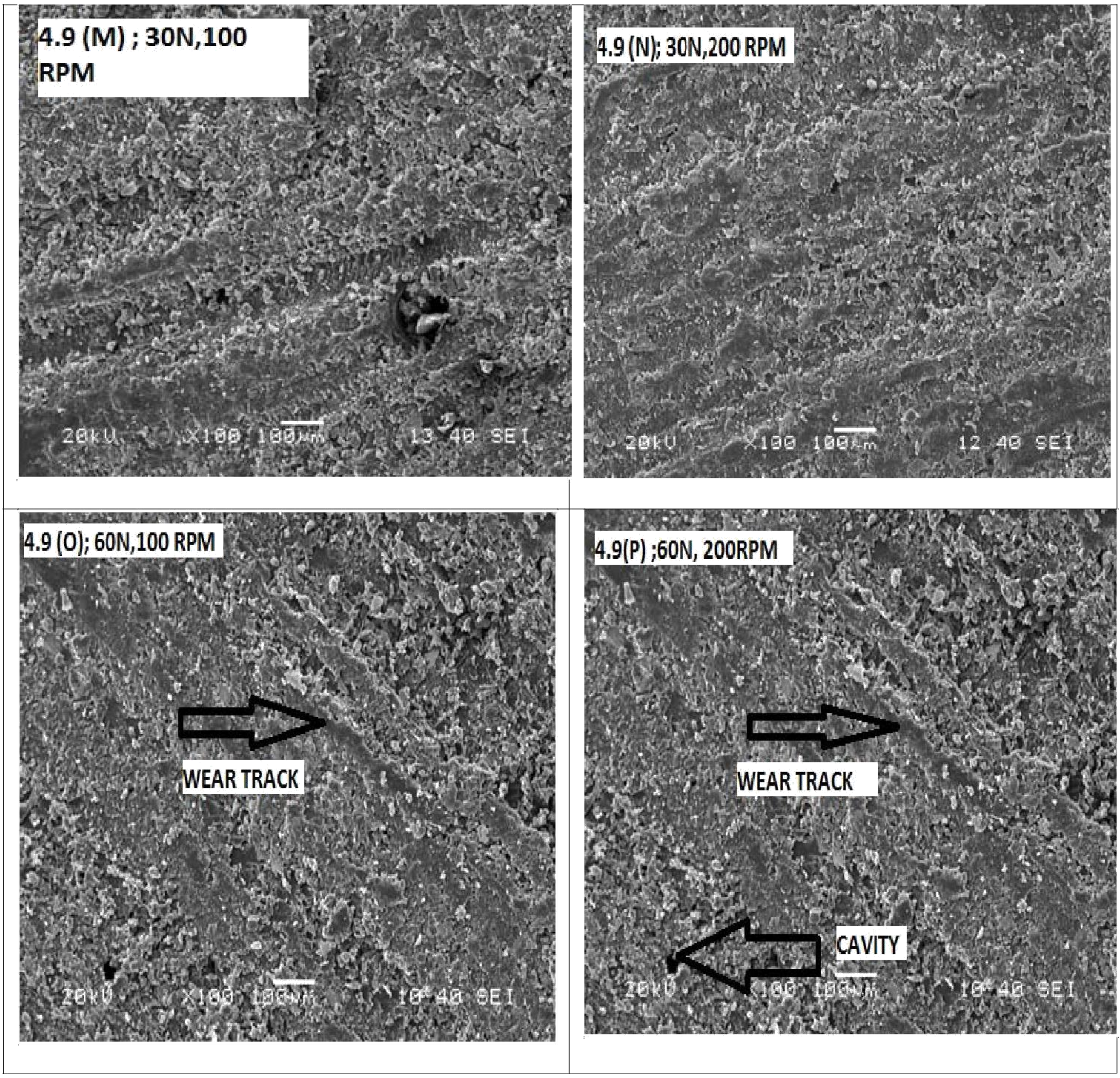


Figure 4.9 (M,N,O,P) ; SEM Microstructure of Wear Track.

From the above scanning electron micrographs (of the wear tested) composite, it is observed that some amount of cavities are formed but good bonding with matrix is observed. From the above image wear track is clearly visible and plastic flow of epoxy also takes place.

CHAPTER – V

CONCLUSION

5. Conclusions

On the basis of present investigation, following conclusion can be drawn.

1. It is concluded that with increase in proportion of iron powder in the composite, the density is increasing. In iron powder and epoxy resin composition (1:9) density was found to be 1.619 gm/cc and in composition (3:7) it is 2.019gm/cc.

2. The hardness test shows that the composition with higher iron powder possesses higher hardness.

3. With increase in addition of iron oxide powder, compressive strength decreases. The compressive strength of 10% iron powder composition is established to be 224.6Mpa and the compressive strength of 30% iron powder composition is found to be 120Mpa. This may be due to cavity formation and /or improper bonding with the matrix.

4. From wear study it is concluded that, with increase in amount of reinforcement and speed, wear rate increases. Wear rate, frictional force and co-efficient of friction are increasing with increase of time. Wear rate increases with increase in applied load.

5. SEM analysis shows the cavities, pits present in the sliding surface of the samples. Wear tracks are formed due to micro-cutting formed during sliding. Plastic flow of epoxy resin also takes place on the surface.

6. From the dielectric analysis it is concluded that, the dielectric constant decreases with increasing frequency. Tan delta, impedance and capacitance etc. first increases then decreases with increase in frequency.

CHAPTER – VI

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